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# Final Report

# "Scanning Probe Microscope for the Analysis of Thermal and Electrical Properties of Nanolaminates"

AFOSR Grant No. F49620-00-1-0189

Prof. Steven M. George, PI

Dept. of Chemistry and Biochemistry University of Colorado Boulder, CO 80309



#### I. Overview

We received funding of \$144,540 for a special scanning probe microscope (SPM) and associated instrumentation under the Defense University Research Instrumentation Program (DURIP) for the Fiscal Year 1999. We were informed of this funding in February 2000. The funds were made available from March 31, 2000 to March 30, 2001. During this period of time, we purchased the new SPM and related hardware and installed this new instrumentation. We are pleased to report that the new equipment is working extremely well and has made a significant impact on our research effort.

Our research focuses on the fabrication of nanolaminates with atomic layer deposition (ALD) techniques based on sequential self-limiting surface chemistry. We are also concentrating on measuring the properties of nanolaminates. Nanolaminates are multilayered thin film structures with very high interfacial density. These composite multilayer structures can display interesting properties that are not observed in the individual components. These special properties can be optimized by manipulating the thickness and composition of the individual nanolayers. The optimized nanolaminates may have important applications as better protective coatings and thin films with enhanced electrical properties.

We needed the new SPM and related instrumentation to characterize the properties of our nanolaminates. Since the installation of the new equipment, we have begun to characterize the metal/ceramic and ceramic/ceramic nanolaminates that we are fabricating using ALD techniques. Our initial efforts have focused on film growth rate, surface morphology and electrical properties. Our future studies employing the new equipment will expand to thermal and additional electrical characterization.

## II. Acquired Equipment

Using the funding provided by this DURIP grant, we obtained the following instrumentation:

# 1. AutoProbe CP Research Scanning Probe Microscope

The AutoProbe CP Research Scanning Probe Microscope (SPM) was obtained from ThermoMicroscopes in Sunnyvale, California. This SPM was equipped with atomic force microscopy (AFM), scanning thermal microscopy (SThM) and conducting atomic force microscopy (C-AFM). The total cost of this SPM was \$121,499.

#### 2. Dektak3 Surface Profiler

The Dektak3 Surface Profiler was obtained from Veeco Metrology Group in Santa Barbara, California. This surface profiler can accurately measure step heights for the quantitative measurement of thin film thicknesses > 100. The total cost of this surface profiler was \$23,041.

#### III. Research and Educational Use of Equipment

The new SPM and surface profiler have already greatly impacted our AFOSR-sponsored research and education. The new SPM was installed in August 2000. Since that time, the new SPM has been involved in much of our AFOSR-sponsored research on nanolaminates. The AFM capabilities of the new SPM have been used extensively to characterize all the thin films and nanolaminates that we have deposited using ALD techniques. In particular, the new AFM capabilities were critical to a study of surface roughness that we performed for Al<sub>2</sub>O<sub>3</sub>/ZnO nanolaminates. We discovered that the surface roughness could be controlled by varying the thickness of the individual nanolayers in the Al<sub>2</sub>O<sub>3</sub>/ZnO nanolaminates.

We have also utilized the new conducting AFM (C-AFM) capabilities of the SPM to study ultrathin dielectric films. The C-AFM can perform current-voltage (IV) and capacitance-voltage (CV) measurements with ~1 micron resolution. These IV and CV measurements will be extremely valuable to characterize defects in thin films and nanolaminates. To date, the C-AFM measurements are still not reproducible because of difficulties with the tips. We have explored a number of tip vendors. The doped-diamond coated silicon tips have yielded the best results. Unfortunately, reproducible results may depend on further improvements in the tip technology.

We have not yet utilized the scanning thermal microscope (SThM) capabilities of the new SPM. This technique has the potential to measure the thermal conductivity of our thin films and nanolaminates. We hope to relate nanolaminate geometry with thermal conductivity to establish structure/property relationships for nanolaminates. These structure/property relationships may have important consequences for the design of superior thermal barrier coatings.

The new surface profiler has also been extremely valuable to our research effort. The surface profiler yields very accurate measurements of step heights for thin films. By masking a starting substrate prior to deposition, the surface profiler allows the deposited film thicknesses to be measured with ~10 accuracy for films thicker than 100 . This simple measurement allows growth rates to be established for ALD thin film growth. We have made extensive use of the surface profiler for this purpose. In addition, the surface profiler has also been employed for characterizing the surface roughness of substrates over dimensions up to 1 mm.

Both the new SPM and surface profiler have provided a superb educational experience for the undergraduates, graduate students and postdoctoral associates who have used these instruments. All the personnel have become acquainted with AFM techniques. The technique development of C-AFM has provided a challenging educational experience. Likewise, the technique development of SThM is also expected to provide additional educational experiences. The surface profiler has become the main technique employed by an undergraduate who is performing his senior thesis in the research group. His project is examining the deposition of oxides on polymers using ALD techniques. This work could become the basis of additional research in the area of inorganic-organic composites.

Program Name: Photonics Research Experiences for Undergraduates

Amount requested: \$40,000

### Proposed internship program summary:

The Photonics Research Experience for Undergraduates (Photonics REU) Program is designed to expose Colorado residents, who are attending an undergraduate institution, to the field of photonics, optics, and optoelectronics. As a result, we expect some of these Colorado undergraduate students will be motivated to pursue graduate studies in photonics, optics, or optoelectronics at one of Colorado s research universities.

Under this program, a university researcher applies for a \$5,000 grant that will fund an undergraduate student full-time for the summer, or half-time for a semester, on a research project that involves optics, photonics, or optoelectronics. The grant is intended to pay primarily for the student s salary. At the end of the grant, the student must submit a summary report of the research results to CPOP.

## Internship matching funds:

In FY00-01 the universities participating in the Photonics REU program contributed approximately \$36,560, while CCHE contributed \$40,000. The university contribution came from: (1) waived indirect costs (\$18,000), and (2) supervision of interns by faculty members (\$18,560). We anticipate matching funds at a similar level in FY01-02.

### Estimated number of students:

We expect to be able to accommodate up to 8 undergraduate students.

#### **Selection Process:**

In order to qualify for the Photonics REU program, a faculty member must:

- (1) Be employed by Colorado School of Mines, Colorado State University, University of Colorado, or University of Denver.
- (2) Agree to provide a report at the end of the grant summarizing the project.
- (3) Propose a research or technology development program in the area of optics, photonics, or optoelectronics.
- (4) Staff the project with an undergraduate student who is a Colorado resident (as classified by the student s institution).

Application to the program involves describing the research project and the undergraduate student s role in the project. The proposal must indicate who will supervise the student, and must show how the student will develop an appreciation of optics, photonics, and optoelectronics as a result of this project. Proposals are to be no more than 2 pages in length. A budget for the project shall be attached as a separate document.

The Governing Board of CPOP, using the following criteria, will evaluate proposals:

- (1) project within the area of optoelectronics, optics, or photonics.
- (2) potential for motivating an undergraduate student to pursue graduate studies in optics, photonics, or optoelectronics at a Colorado university,
- (3) technical merit.

If the university P.I. has had a previous CPOP grant, the performance on that grant may be used as a factor in evaluating the Photonics REU proposal.

#### Mentoring Plan:

The undergraduate students will work in university research groups where they will interact with graduate students, post-docs, and faculty members. In addition to seeing how a photonics research group works, they will have responsibility for a portion of the project. A faculty member will be responsible for the photonics experience for each of the interns.